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Electron Stimulated Desorption-Ion Angular Distribution (ESDIAD): A Method for  
Imaging Chemical Bond Directions and Thermal Disorder in Adsorbed Species

by

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Pittsburgh, PA 15260

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| 20. ABSTRACT (Continue on reverse side if necessary and identify by block number)<br>The ESDIAD method and its application to typical systems is described.                                  |                       |  |  |

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**ELECTRON STIMULATED DESORPTION-ION ANGULAR  
DISTRIBUTION (ESDIAD): A METHOD FOR IMAGING  
CHEMICAL BOND DIRECTIONS AND THERMAL DISORDER IN  
ADSORBED SPECIES\***

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**I. INTRODUCTION**

The measurement of the molecular properties of adsorbed species on single crystal surfaces forms one of the major goals of surface science research. A number of measurement techniques permit the characterization of chemical bonds at surfaces using different physical principles. The technique considered here involves the electronic excitation of the bond causing its rupture- the measurement of the ejection angle of the fragment species produced may be used to estimate the azimuthal and polar orientation of the bond being broken. In addition, certain aspects of the dynamical behavior of the adsorbed species may also be observed from the temperature dependence of the desorbing particles' angular distribution.

The technique which provides this capability to measure bond directionality and adsorbate dynamics is ESDIAD- electron stimulated desorption ion angular distribution. The ESDIAD method was invented in 1974 [1], based on pioneering

studies of electron stimulated desorption (ESD) done almost 10 years earlier [2,3]. The early studies [2,3] were not concerned with the measurement of the angular distribution of desorbing particles, and the discovery of the ESDIAD phenomenon [1] led a number of workers to successfully employ the technique for investigations of the chemisorption of small molecules on metal surfaces. Recently, the technique has been extended to semiconductor surfaces with great success. A recent review of ESD and ESDIAD has been published [4]. About 1000 papers have been written on ESD, and ESDIAD has been employed in about 10% of these papers. The special application of ESDIAD to the study of bond dynamics has also been reviewed recently [5]. This article will briefly discuss the principles which govern ESDIAD; it will illustrate how the measurements are made, and two examples of the application of ESDIAD will be given.

## II. THE ESDIAD PHENOMENON

Figure 1 shows a potential energy diagram for an adsorbed atom, A, on a single crystal surface, designated S. The probability distribution for locating the particle in its ground electronic state somewhere on its adsorption site is schematically indicated by the gaussian-shaped distribution function as designated by the vibrational wavefunction squared,  $\psi^2$ . An electron,  $e^-$ , incident on the particle will cause an electronic excitation to an upper state and in some cases this will lead to the particle's arrival at a repulsive potential energy surface which ejects the  $A^+$  ion from the surface as shown in the figure. The vertical excitation is a rapid Franck-Condon process, exciting the particle from a particular point in the ground state distribution function. If all of the adsorbed A atoms which contribute to the  $A^+$  production originate from an ensemble of identical sites on the single crystal surface, then the measurement of a large number of ESDIAD

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events will statistically sample the entire ground state distribution function. Thus, the spatial distribution of desorbing particles will reflect the probability distribution of adsorption positions on the identical sites. In particular, the bond angles contained in the distribution function will be sampled in the ESDIAD pattern, and the most probable measured ejection direction will correlate with the most probable bond angle for the species in its chemisorption state. Thus ESDIAD may be used to image the bond angles of surface species by imaging the directions of ejection of fragments of the surface species produced in the electronic excitation. In addition, the angular width of the  $A^+$  ESDIAD pattern contains information about the bond dynamics which are characteristic of the adsorbed species. ESDIAD therefore is a powerful technique for observing the most probable bond angle as well as the spread of bond angle due to zero point and thermal excursions of the particle in its motion to-and-fro on its adsorption site. The measured angles of ejection of ions must be corrected for final state effects which include deflections by attractive image effects between the ion and the substrate from which the ion is escaping [6]. Also, angle and velocity dependent neutralization effects must be considered in correcting the measured ejection angles to determine the true bond directions [6,7]. In addition, in recent years it has been found that neutral metastable electronically excited particles are also produced in ESD, and in some cases these may be detected by the ESDIAD apparatus [8].

### III. THE ESDIAD MEASUREMENT TECHNIQUE

The original ESDIAD method [1], which was employed from 1974 until 1984, involved the use of a phosphor screen detector for the measurement of the positive ion angular distribution originating from oriented chemical bonds on single crystal surfaces. One exception to this statement is noted, namely the use of

a movable channeltron detector for mapping ion angular distributions as developed by Niehus [9]. The use of phosphor screen detectors has now largely been superseded by particle counting methods. A schematic diagram of the particle counting ESDIAD apparatus at the University of Pittsburgh is shown in Figure 2. In this apparatus, an electron beam is incident on a single crystal containing an adsorbed overlayer. The positive ions produced by excitation of the surface species desorb from the crystal in directions closely related to the direction of the chemical bond being dissociated, and they travel toward the pair of hemispherical grids in front of the single crystal. From the second hemispherical grid, the positive ions travel to a planar grid and are accelerated by a second planar grid at -1.2 kV. Each positive ion enters the front surface of the first microchannel plate (MCP), and, by secondary emission, generates a pulse of about  $10^6$  electrons which exit the second microchannel plate and are collected on a resistive anode (Surface Science Laboratories, No. 3394SD). The electron pulse is spatially correlated with the original ion pulse. This pulse of electron charge expands radially as a ring of charge on a properly edge-terminated resistive anode, and, since the local intensity of expanding charge decreases as  $1/r$ , the pulse height arriving at the four corners can be used to calculate the coordinates of the point of impact of the electron pulse (the centroid). The pulses at the four corners are capacitively coupled into four preamplifiers, A, B, C, and D. These amplified pulses then travel into the position computer (Surface Science Laboratories 2401). The position computer then generates a pair of pulses whose heights are proportional to the x and y coordinates of the impact point on the resistive anode. The x and y pulses are then sent to a pair of analog-to-digital converters that form the input to a Nuclear Data 76B multichannel analyzer (MCA). The MCA has the capability of displaying the three dimensional ion angular distribution in a projection on the screen that shows the number of events as a function of x, y

position. This apparatus is capable of yielding far more visual and quantitative information than previous photographic or movable detector systems, with an ultimate resolution of about  $0.3^\circ$ .

All previous photographic and movable detector ESDIAD systems have suffered from the presence of a background effect caused by soft X-rays emitted from the crystal during bombardment by the electron beam. This background can be measured on the clean crystal, and accurately removed by subtraction from the digital data [10].

By reversing the grid biases, we can employ the angle-resolved, hemispherical retarding field analyzer as a digital LEED apparatus in which background X-ray effects can also be removed by subtraction [10].

Thus, the digital ESDIAD/LEED apparatus permits one to accurately compare the angular distribution of positive ions fragments or certain electronically-excited species with the orientation of the crystallographic axes of the single crystal substrate. In general, a simple geometrical relation between crystal structure and ion angular distribution is observed, showing that chemical bond directions in adsorbed species are closely controlled by the structure of the underlying substrate [4].

In comparison to the older photographic ESDIAD method, the dynamic range of the digital detection system is not limited by the behavior of a photographic emulsion, and mathematical routines may be conveniently employed to measure angular profiles more thoroughly than is possible otherwise.

#### IV. SELECTED EXAMPLES OF ESDIAD MEASUREMENTS ON METAL AND SEMICONDUCTOR SURFACES

##### A. Chemisorption on a Metal Surface

The predominant ion fragment produced by electron bombardment ( $V_e = 1000$  V) of chemisorbed CO on Ni(110) is the  $O^+$  ion. The  $O^+$  ion is ejected along the C-O bond direction and this direction can be used to determine the angular direction of the C-O bond in the surface species. The Ni(110) surface is interesting as a substrate for chemisorption because it consists of alternate rows of surface nickel atoms separated by open channels.

Figure 3 shows the ion angular distribution from CO chemisorbed on Ni(110) under various conditions. Figure 3A shows the ion angular distribution for the CO layer at a coverage of one-half monolayer. This pattern is superimposed on top of the X-ray background. Figure 3B shows the same pattern with the X-ray background removed by subtraction. A normally-oriented distribution of positive ions is observed. The LEED structure [11] corresponding to a coverage of 5/8 monolayer of CO is shown in Figure 3C. Figure 3D shows the ion angular distribution obtained at a full monolayer coverage, again superimposed on the X-ray background, which is removed in Figure 3E. Here one sees that the normally-oriented ion angular distribution has been replaced by a two-beam distribution. The two beams of ions are oriented in a plane accurately along the (001) direction. The LEED pattern corresponding to this ESDIAD pattern is designated (2x1) p1g1, and the structure of the crowded overlayer is shown in Figure 3F. It should be noted that in order to produce the crowded p1g1 structure it is necessary to pack the CO molecules in a zig-zag pattern, alternately left and right of the uppermost Ni atom rows [11,12]. The ESDIAD results



indicate that the C-O bond axis tilts to the left and right as the plgl surface phase forms. We have measured the angle of tilt and find it to be  $19^\circ$  from the normal, in agreement with the work of others [13].

### **B. Chemisorption on a Semiconductor Surface**

The application of ESDIAD to the study of adsorbates on semiconductor surfaces is just beginning. Understanding adsorbate bonding and dynamics on semiconductor surfaces is of great importance because of the technological need to control chemical processing methods for electronic device fabrication. Semiconductors, in contrast to metals, are internally bonded by directed covalent bonds, and unsatisfied valencies (dangling bonds) project upward from the surface. It is here that adsorbates form highly localized chemical bonds to the surface. The first ESDIAD studies on a covalent surface, Si(100), concerned the adsorption of fluorine [14]. More recently, -Cl groups produced by  $\text{Cl}_2$  chemisorption near 500 K on Si(100)-(2x1) have been investigated with ESDIAD [7,15]. A four-beam  $\text{Cl}^+$  pattern is observed at 120 K as shown in Figure 4b and 4c. These  $\text{Cl}^+$  beams originate from Si-Cl covalent bonds, which are inclined from the surface normal in four specific directions at an angle of  $25 \pm 4^\circ$  from the surface normal [7]. These four directions correspond to the orthogonal Si-Si surface dimer bond directions exposed on alternate terraces of Si(100); the steps and terraces are caused by slight misalignment of the crystal, and a symmetric dimer site in one domain is shown in Figure 4a.

The ESDIAD patterns shown in this article are mathematically smoothed patterns of digital data, where the total number of ion counts collected is generally near  $10^6$ . An actual digital ESDIAD pattern, corresponding to the  $\text{Cl}^+$  pattern of Figure 4, is shown in Figure 5. Here, each point shown represents the uppermost

point in a series of points stacked within a single channel in the collected angular distribution. Each channel is separated from its neighbors by about  $0.3^\circ$ . It may be seen from the figure that the natural width of the four  $\text{Cl}^+$  beams is much larger than the instrumental resolution of individual ion arrival angles (channel separation). The high angular resolution has permitted detailed observation of the shape change in the ESDIAD patterns as the surface is heated [15]. Such changes are used to characterize the vibration-dynamic behavior of the adsorbed species, permitting one to visualize the thermal motion of the adsorbed species on its adsorption site [5,15].

## V. FUTURE DIRECTIONS

It has been shown that ESDIAD is capable of imaging chemical bond directions for small molecules or atoms chemisorbed on single crystal surfaces. In addition, although not explicitly discussed in detail in this article, the ESDIAD method permits one to take dynamic snapshots of the bond orientations in an ensemble of adsorbate species and therefore to directly observe the angular range of thermally excited motions which contribute to thermal broadening of the ESDIAD pattern. This capability to image the thermal motion of adsorbates and to detect anisotropies in the thermal disorder is currently a unique capability of ESDIAD in the arsenal of surface measurement techniques of the surface scientist. This capability is akin to the measurement of the thermal ellipsoids of vibration for atoms in a crystal as currently practiced in x-ray crystallography. The ability to observe in real space the thermal excursions of an adsorbed species is of importance in giving new molecular level understanding of surface phenomena. For example, the factors which are responsible for preferential surface migration directions and for preferential chemical reaction geometries for adsorbates on

model single crystal surfaces may be related to the dynamical on-site behavior of individual chemisorbed species. Thus, ESDIAD is expected to contribute key information about fundamental geometrical and molecular dynamical factors governing surface chemistry. Much of this awaits future research.

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## FIGURE CAPTIONS

**Figure 1.** Schematic potential energy surfaces for an adsorbed atom showing its excitation to a repulsive surface. Neutralization processes are effective in returning many  $A^+$  ions to lower states. One of two generalized coordinates parallel to the surface is designated  $q_{\parallel}$ . The dynamical spread of the atom's coordinates is indicated by the probability distribution in the ground state potential well. S, surface; A, adsorbate atom.

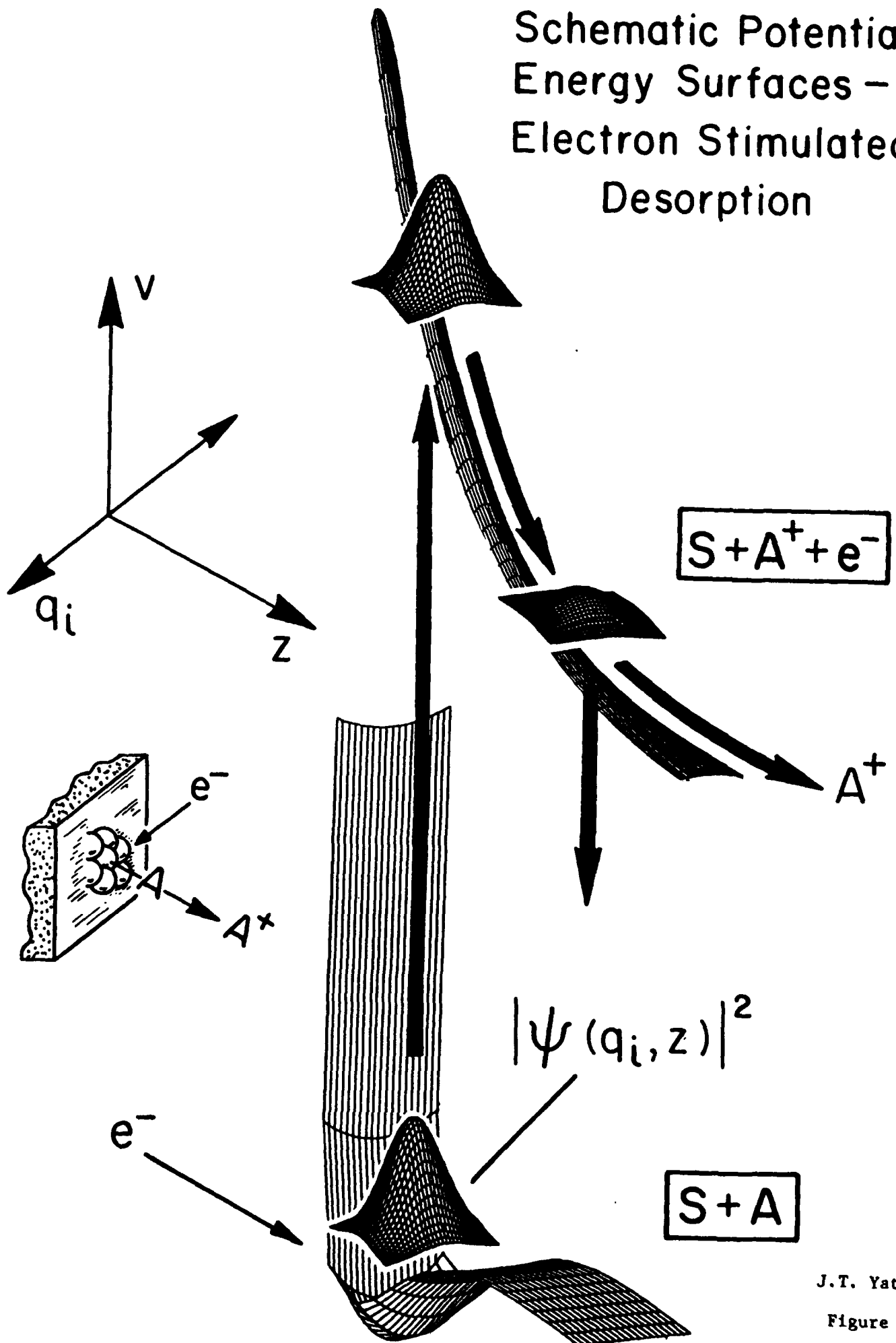
**Figure 2.** Digital ESDIAD apparatus for measurement of angular distributions of species originating from electron stimulated desorption from a single crystal surface. An angular distribution pattern is displayed on the multichannel analyzer, MCA. By reversing potentials on the grids, the analyzer may be used in the low energy electron diffraction (LEED) mode, permitting the ion angular distribution to be compared to the lattice orientation of the single crystal substrate. ADC, analog-digital converter.

**Figure 3.** Comparison of digitally acquired  $O^+$  ESDIAD patterns for chemisorbed CO on Ni(110). Panels A and D are the raw ESDIAD data detected for the two indicated surface coverages,  $\theta_{CO}$ . Panels B and E have the background subtracted and are mathematically smoothed. Panels C and F show models of the adsorbate configurations postulated on the basis of LEED studies. The tilting CO molecules produced by surface crowding are confirmed by the comparison of ESDIAD and LEED measurements.

**Figure 4.**  $\text{Cl}^+$  ESDIAD pattern from a full coverage Cl layer produced on  $\text{Si}(100)-(2 \times 1)$ . Panel a shows a symmetric silicon dimer site containing two Si-Cl surface groups. The Si-Cl bonds are inclined  $25^\circ$  from the normal and are thermally disordered as schematically represented by the drawing in which a spread of Si-Cl bond directions is indicated. Since the  $\text{Si}_2$  dimer sites coexist in orthogonal domains on the crystal surface because of a small step density, four characteristic Si-Cl bond directions coexist and are imaged by the ESDIAD method as shown in Panel b. A contour diagram of the four beam pattern is shown in Panel c, where the ESDIAD measurement is made at a crystal temperature of 120 K.

**Figure 5.** Actual digital data for  $\text{Cl}/\text{Si}(100)$ , showing the density of measurement channels for  $\text{Cl}^+$  data acquisition. About one million  $\text{Cl}^+$  trajectories are stored in this pattern. The damage to the surface by ESD during this measurement was less than 1% of a monolayer.

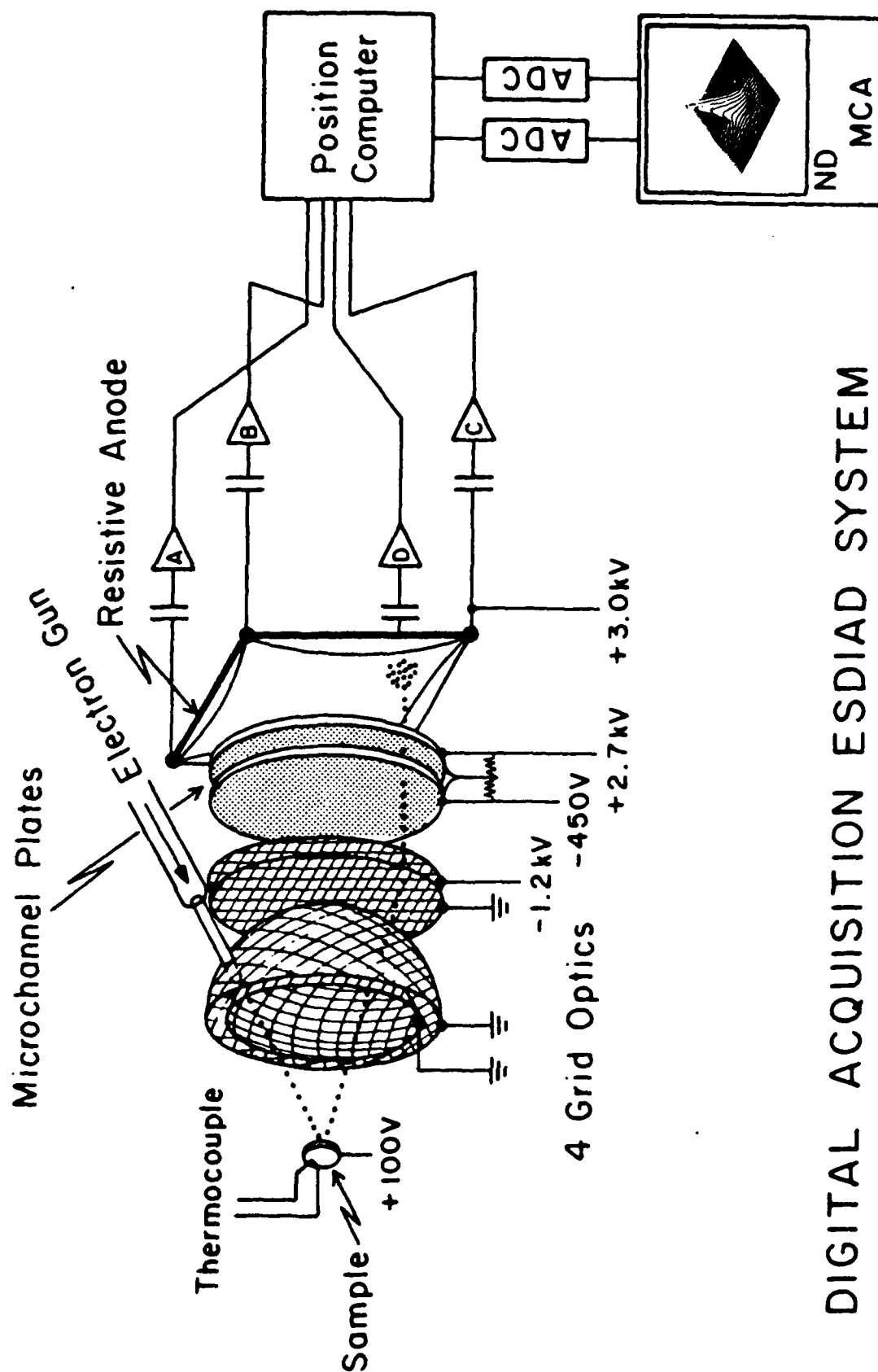
# Schematic Potential Energy Surfaces – Electron Stimulated Desorption



J.T. Yates, Jr.

Figure 1



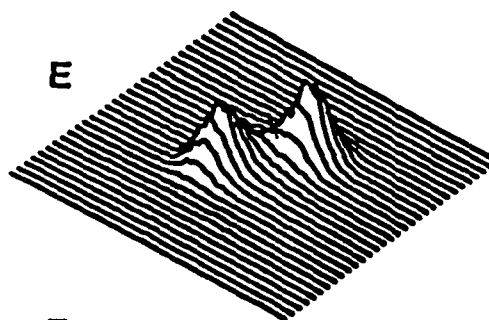
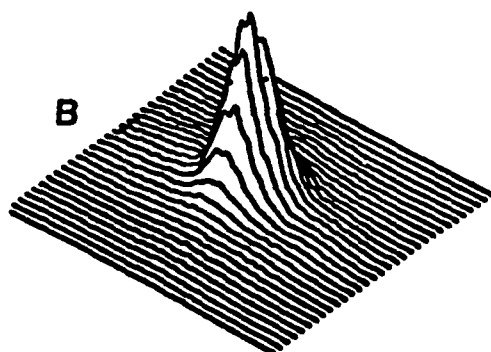
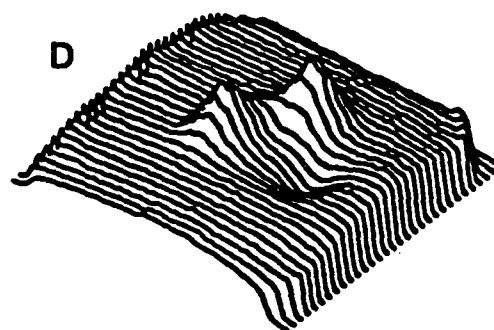
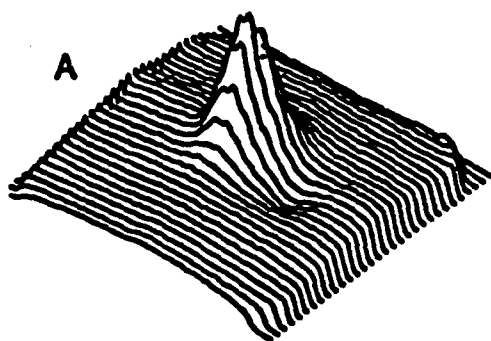


DIGITAL ACQUISITION ESDIAD SYSTEM

# DIGITALLY ACQUIRED ESDIAD FOR CO-Ni(110)

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$\theta_{\text{CO}} = 1.0 \text{ CO/Ni}$

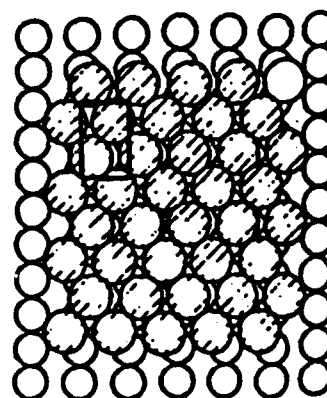
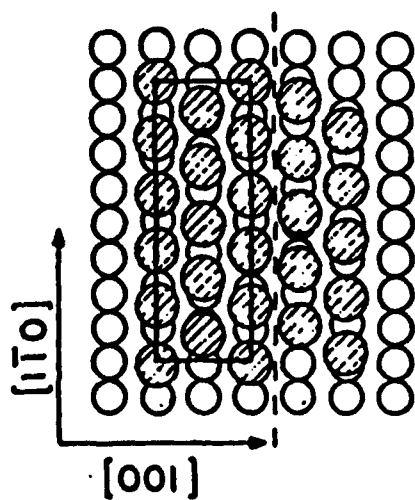


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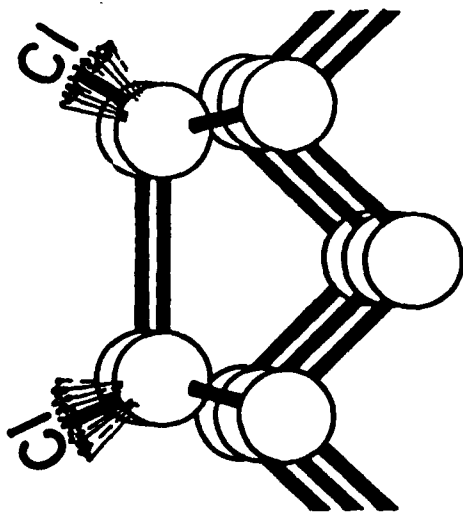
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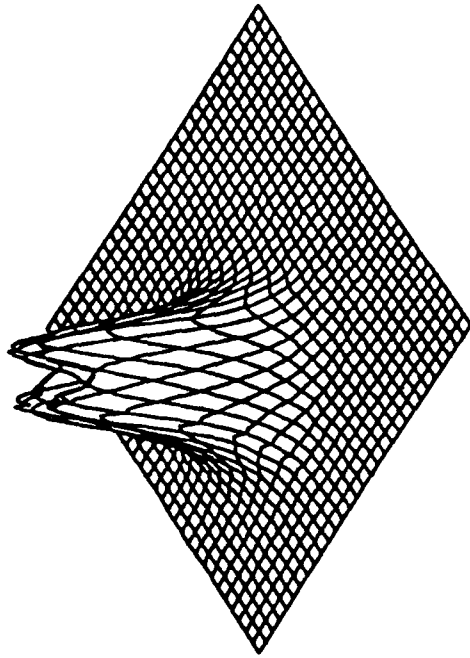


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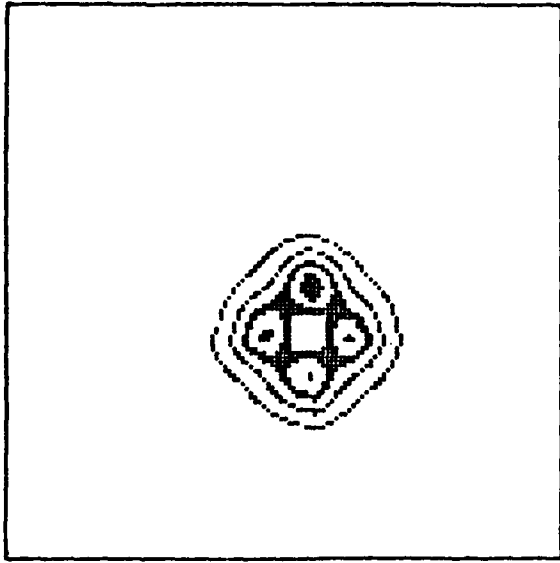
Figure 3



a.

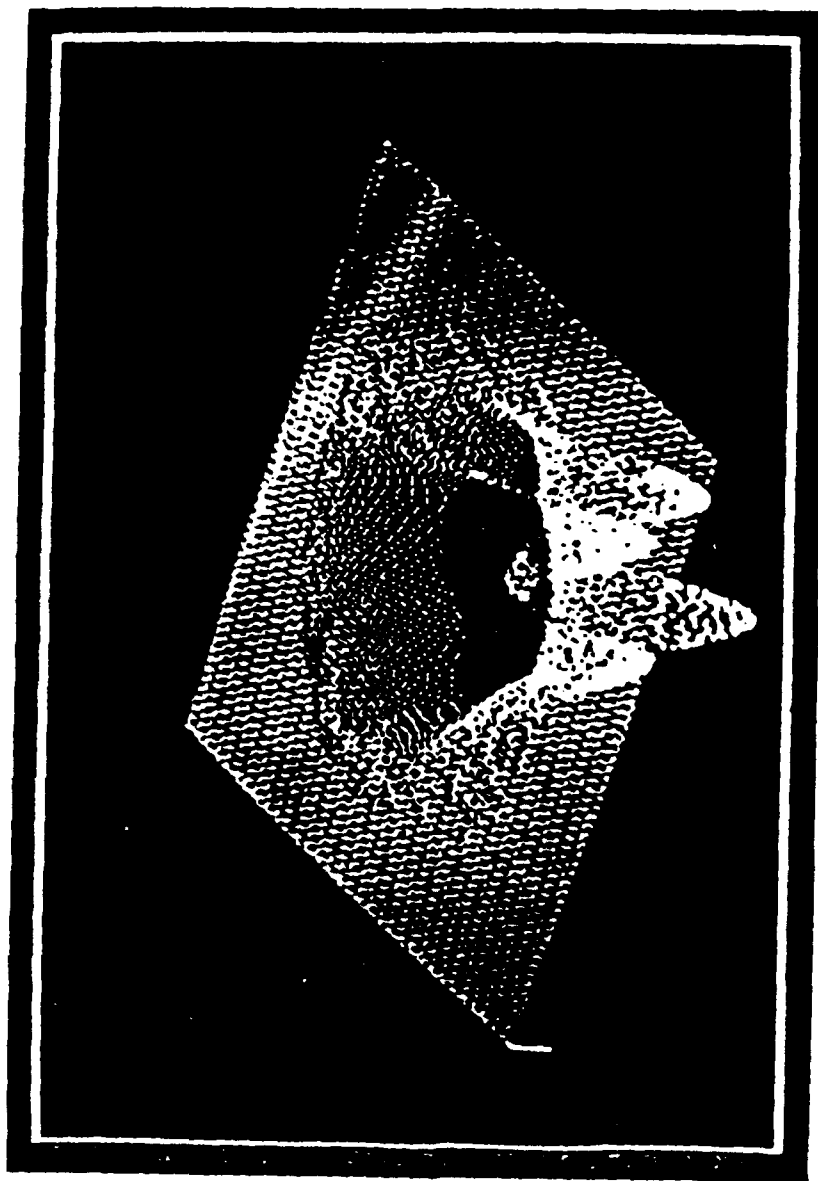


b.



120 K

c.



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Figure 5

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